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TECHNICAL REPORT

RELIABILITY OF M256 CHEMICAL AGENT DETECTOR KIT AT EXTREME ENVIRONMENTAL TEMPERATURES

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February 1988



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FOREWORD

This study was prepared in compliance with the requirements of RDTE Project 1M465710D049, and deals with the reliability of the M256 Chemical Agent Detector Kit under potential operational environments. A brief description of the chemical reactions involved in the detection mechanism is included for general information.

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1. INTRODUCTION

In battles involving chemical warfare (CW) agents, detection and identification of the agents in the environment is essential. Although protective garments and masks can provide soldiers with adequate protection, they drastically hamper the soldiers' normal operations. It is therefore advisable to determine whether CW agents are present in the environment so that the soldier can assume a protective posture appropriate for the situation. The current method of detecting chemical agents is by the use of the M256 chemical agent detector kit (M256 Kit), which identifies blister, nerve, and blood agents.

The M256 kit is crucial for the operation of the combat force in a chemically contaminated environment. Since this device is used primarily as an "all clear" indicator, if it fails to detect the agents present in the environment, casualties can ensue. On the other hand, a false positive indication will cause the needless burden of assuming a protective posture that will reduce the efficiency of the soldier. Also, since exposure for long periods to chemicals in sublethal amounts can be hazardous, periodic monitoring using the M256 kit is essential following a chemical attack.

In compliance with the requirement of RDTE Project IM465710D049, this study was undertaken to investigate the effect of exposure of the M256 kit to extreme climatic conditions on its sensitivity and reliability in detecting CW agents. Such information was warranted mainly because the climatic condition and the terrain at which the U.S. soldiers may have to confront a chemical attack remains uncertain. Since detection is based on chemical reactions between the CW agents and the specific reagents provided in the 'ready to use' ampoules of the kit, a brief review of the chemical reactions involved is given in this report. Detailed information on the chemistry and the engineering aspects of the kit are available in References 1 and 2.

2. EVALUATION OF THE M256 KIT

2.1 GENERAL DESCRIPTION

The M256 kit has two components, a booklet of M8 paper (ABCVGH-M8), which detects liquid chemical agents, and a sampler/detector, which is designed to detect and identify agent vapors. This report will discuss only the vapor detection mechanism. The sampler/detector part of the kit has been designed to detect three main classes of CW agents. Table I lists the agent vapor that can be detected by the device.

The packaged reagents are premixed in finger-crushable glass ampoules and the individual tests are carried out at designated spots on the sampler discs. Figures 1 and 2 show the design of the kit. The layout of the sampler/detector is noteworthy in that special care has been taken to avoid interference between the tests for various agents. Since the detection mechanism of the kit is based on chemical and enzymatic reactions, a brief examination of the reactions is pertinent.

TABLE 1. Chemical Warfare Agents Detectable by the M256 Kit

Agent	Detectable Concentration (mg/m ³)
Nerve	
VX	0.10 ± 0.02
G	0.05 ± 0.01
Blood	
Hydrogen Cynanide (AC)	9.0 ± 2.0
Cyanogen Chloride (CK)	9.0 <u>+</u> 2.0
Blister	
Lewisite (L)	9.0 ± 5.0
Mustard (H)	2.0 <u>+</u> 1.0
Phosgene Oxime (CX)	

TM 3-6665-307-10

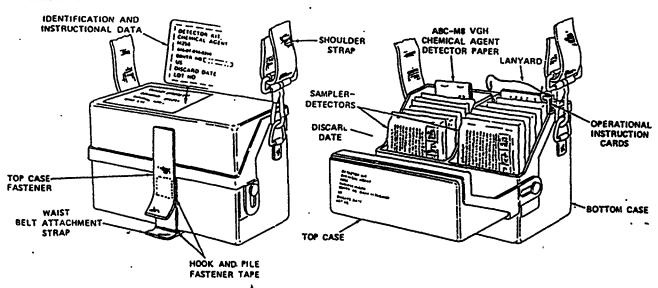


Figure 1. M256 Chemical Agent Detector Kit (front view). Reproduced from TM 3-6665-307-10.

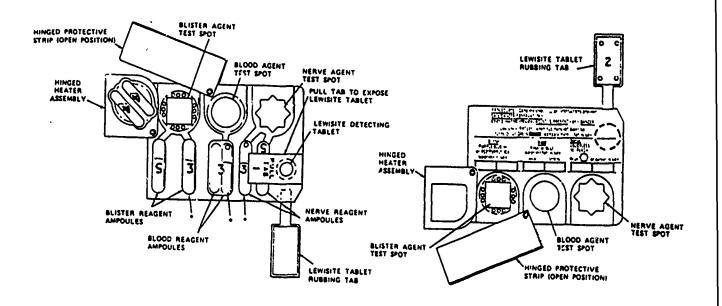


Figure 2. Sampler - Detector (protective bag removed). Reproduced from TM 3-6665-307-10.

2.2 CHEMICAL REACTIONS

a. Nerve Agents

The M256 kit detects G and V agents by taking advantage of the competition of these agents for the active site of the enzyme acetylcholinesterase with the substrate 2,6-dichlorophenol acetate (2,6-DIPA). In the absence of nerve agent, acetylcholinesterase (immobilized on a filter paper spot) converts 2,6-DIPA into 2,6-dichlorophenol, a compound that is blue in color. If either G or V agent is present it will tie up the enzyme, which will then not be available to react with 2,6-DIPA to form the blue color. The test is carried out on the filter paper spot. Tris buffer and 2,6-DIPA are provided in two separate ampoules.

b. Blood Agents

A pink or blue color complex is formed when CK (cyanogen chloride) reacts with 4-benzylpyridine and barbituric acid. If the agent AC (hydrogen cyanide) is present, it is converted to CK by the sodium hypochlorite solution provided in one of the ampoules. In the sampler/detector, the reaction test spot is impregnated with barbituric acid, and the 4-benzylpyridine is provided in an ampoule. When the reagents are mixed, the color change indicates the presence of CK or AC in the air. In the absence of the agents, the test spot remains colorless.

c. Blister Agents

Mustard gas (H) can be detected because of its reaction with a methanolic solution of DB-3 [4-(4'-nitrobenzyl)pyridine] in the presence of a catalyst, mercuric cyanide. The product of this reaction then reacts with potassium carbonate to form an intense blue-purple color. A similar reaction occurs when phosgene oxime (CX) is present, the final color being pink or red. Both of these reactions are faster at elevated temperatures; therefore, a chemical heating component (cupric chloride and aluminum powder) is included in the kit.

d. Lewisite

The test for lewisite involves the reaction between lewisite and Michler's thicketone [4,4-bis (dimethylamino)thiobenzophenone]. The reagent is immobilized in an inert carrier and extruded into a crayon. The crayon when smeared into a white background turns olive-drab green when lewisite is present.

2.3 ENVIRONMENTAL TESTING

The design and sensitivity of the chemical reactions of the M256 kit have been subjected to a number of tests, and tests were used in making modifications. The problems encountered during the developmental stages of the kit are summarized by Turner et al. (Ref. 2). These aspects are beyond the scope of this report and therefore not discussed.

In a study conducted at Yuma Proving Ground (Ref. 3), M256 kits were subjected to a 90-day field-carry test; the samplers were then challenged with either CW agent or a control substitute at hot and humid conditions. Tests to measure the responses to each agent were then carried out. The results are given in Table 2. The samplers responded properly to all CW agents. All responses were negative for the control tests. From this test it was concluded that at intermediate hot-dry (52°C) conditions, the 1256 kit is reliable.

In a separate study conducted by U.S. Army Dugway Proving Ground (Ref. 4), 90-day field-carried kits (at Tropic Test Center) were exposed to hot-wet conditions. The results are presented in Table 3. Sampler response was examined when the temperature was 35°C and the relative humidity, 74 percent. All of the samplers showed proper responses indicating that at this environmental condition, the M256 kit is reliable.

A test on the operation of the M256 kit was also carried out in cold weather conditions (the temperature ranged from -38 to 9° C) (Ref. 5). It was noticed that false-positive nerve agent responses could occur when the atmospheric temperature was -9° C or below. Several ampoules were frozen when stored at -20° C. Although at cold temperatures the immobilized acetylcholinesterase (on the reaction pad) was stable, a decrease in the enzyme activity may be expected.

2.4 OPERATION OF M256 KIT UNDER EXTREME TEMPERATURE

The reliability of the M256 kit appears to be good at high environmental temperatures. However, one needs to be cautious in using the kit at an extremely hot, dry condition, for example in the Middle East where high winds can be associated with the high temperature. The liquid reagents are essential for the color development (for a period of about 20 minutes); however, wind will cause the reagents to evaporate quickly. This problem may be minimized by placing the kit in an open box protected from direct wind.

As indicated, cold temperatures can adversely affect the M256 kit. The arctic wind can bring the temperature substantially below the freezing point of the reagents and impair the use of the kit.

In the technical manual (TM) for Chemical Agent Detector Kit, M256 (TM 3-6665-207-10-Ref. 5), information concerning low temperature operation is as follows:

"When the temperature is below freezing, the sealed protective bag containing the samples must be warmed (in a shelter, venicle, or within the operator's clotning) until the liquid reagents in the ampoules are no longer frozen. After the bags are opened and examined, the samples are warmed again in the operator's clotning without contaminating it. If the temperature is below $50 \text{ F} (10\,^{\circ}\text{C})$ wait for five minutes before reading the color. Wait for two minutes after conducting tests to observe color comparison."

The recently revised Til (TM 3-6565-307-10-Ref.7) also instructs that frozen reagents have to be thawed completely before the kit is used.

PERFORMANCE OF THE M256 KIT IN A DESERT ENVIRONMENTA TABLE 2.

90-Day		Agent	Tempera-	Relative	Proportion	Reliability of Response (%)	Response (%)
Carry Test Environment	Agent	Conc. (mg/m³)	ture (°C)	Humidity (%)	Kesponding Properly	50% contidence Interval	Sub confidence Interval
Yuma Proving	89	0.011	52	18	27/27	97.4-100	91.8-100
Ground	×	90.0	52	19	12/12	97.4-100	91.8-100
	9	2.0	52	19	72/72	97.4-100	91.8-100
	č	2.0	52	53	72/72	97.4-100	91.8-100
	AC	8.8-9.0	52	23	27/27	97.4-100	91.8-100
	۔	4.0	52	23	12/12	97.4-100	91.8-100
	Blanks	None	52	20	12/12	97.4-100	91.8-100

aThe samplers were challenged under the indicated temperature and relative humidity for 10 minutes. Each reaction was carried out to examine the performance of the kit. The data are taken from Zylstra (Ref. 3).

PERFORMANCE OF THE M256 KIT IN A TROPICAL ENVIRONMENT TABLE 3.

90-Day Carry Test Environment	Agent	Agent Conc. (mg/m³)	Tempera- ture (°C)	Relative Humidity (%)	Proportion Responding Properly	Reliability of Response (%) 50% Confidence 90% Confide Interval	Response (%) 90% Confidence Interval
Tropic Test	68	90.0	35	74	72/73	97.4-100	91.8-100
center	×	0.10	35	75	72/72	97.4-100	91.8-100
	НО	2.5	35	72	72/73	97.4-100	91.8-100
	č	3.0	35	74	17/17a	96.0-100	87.3-100
	AC	11.0	35	74	72/73	97.4-100	91.8-100
	1	11.0	35	74	75/52	97.3-100	91.5-100
	Blanks	None	35	71/73b	27/27	97.4-100	91.8-100

NOTE: After being carried for 90 days in a tropical environment, the samplers were exposed to the indicated temperature and humidity in a chamber and challenged with the agents for 10 minutes. Tests were carred out to examine the performance of each reaction. The data are taken from Zylstra (Ref. 4).

aOnly 17 samplers were tested with CX because of ampoule breakage, which limited the number of available components. ^bBlister components were functional at 71 percent relative humidity, and 11 other components were functional at 73 percent relative humidity.

dowever, the instructions given in a recently published field manual (FM 3-4) on NBC Protection are somewhat misleading. Field Manual 3-4 (Ref. J) emphasizes the need to take special precautions when the M250 kit has to be used in arctic weather where temperatures can be very low. It instructs: "(when the temperature is -15°F (-26°C) or below), the solutions in the capsules freeze, and the solutions will not work even if reheated." This statement places severe restrictions on the use of M250 kit in cold weather. It also contradicts the instructions given in the technical manual (Ref. 7). In the environmental tests carried out prior to the type classification of this kit (Ref. 5), no impairment in the function was noticed following exposure to extreme cold weather. Therefore, the instruction given in FM 3-4 is not correct. It is recommended that appropriate changes be made in FM 3-4 on page B-3.

The technical manual (Ref. 7) cautions that at a night emperature and low humidity (desert conditions), the nerve agent spot may dry up and it is therefore necessary to rewet the spot by squeezing ampoule 3 to deliver a few more drops of the buffer.

At a high temperature and high humidity (tropic) conditions, the color produced by lewisite may be very faint. Therefore, before a judgement is made, this test should be repeated. Also, under tropic conditions a faint blue color may appear even in the absence of the blister agent (Ref. /).

3. CURRENT STATUS OF CHEMICAL AGENT DETECTION

The M256 kit has been subjected to a number of tests and many alterations in the engineering and the detection procedures have been made to make this kit as reliable as possible. The environmental condition that will most impair its function in detecting nerve agents is extreme cold weather. However, because the primary use of the kit is as an "all clear" indicator rather than as a warning sign, the soldiers should have time to bring the kit to a warm temperature before the test is done. Moreover, the testing can be repeated to confirm the results.

A critical evaluation of the usefulness of M250 kit is warranted. As the test is based on the color reactions and visual judgement, human error is possible. This is especially true in night-time operations. In addition, color blind soldiers may find it difficult to use this kit.

Although the Army has accepted the M256 kit, the kit has not met several of the original requirements for the detection of VX, as stated in the material needs (see Ref. 8). Studies have shown that miosis could occur at nerve agent concentrations below the VX detection levels of the kit. Recently, with the use of eel acetylcholinesterase, the sensitivity of the improved version of the M256 (M256Al, during experimental stages the kit was named M256El) can detect concentrations of 6 agent at 0.005 mg/m³ and of VX at 0.013 mg/m³ without making any change in the original design. Table 4 provides some features on the reliability of the M256Al kit. As the data indicate, the M256Al kit should be reliable even when exposed to battlefield contaminants and does not give a false alarm even at extreme temperatures.

TABLE 4 RELIABLILITY OF THE M256A1 (M256E1) KIT

	Test Conditions	Number Trials	Correct Response	Reliability	Confidence
	Blank ^a , ambient (68°F), accelerated aging	50	50	95.5	06
2.	Blank, cold (33°F), accelerated aging				
	a. Response time - 5 min	50	44	80.0	06
	b. Response time - 6 min	50	48	0.06	06
ب	Blank, interferent, accelerated aging				
	a. HC smoke	65	64	94.1	06
	b. Burning brush	48	48	95.3	06
	c. Sevin	. 09	50	95.5	06
4	VX, 018 mg/m ³ ambient (110 F), unaged	33	33	93.3	06

^aNo agent present Data taken from Reference 7.

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REPLY TO ATTENTION OF

RDCB-DPS-RS

MEMORANDUM THRU Director, Edgewood Chemical Biological Center, (RDCB-D/Mr. Joseph Wienand), 5183 Blackhawk Road, Aberdeen Proving Ground, Maryland 21010-5424

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FOR Defense Technical Information Center, 8725 John J. Kingman Road, Ft Belvoir, VA 22060

SUBJECT: Internal Request for Change in Distribution

- 1. This action is in response to an Edgewood Chemical Biological Center (ECBC) Internal Request for a Change in Distribution on documents related to chemical detectors.
- 2. The listed documents in the attachment have been reviewed by ECBC Subject Matter Experts and deemed suitable for the change in distribution to read "Approved for Public Release; distribution unlimited."
- 3. The point of contact is Adana Eilo, ECBC Security Specialist, (410) 436-2063 or adana.l.eilo.civ@mail.mil.

Encl

NANCY CARTER

Acting Security and Surety Manager

Chemical Detector References

- [1] Katzoff, Lionel and Razulis, Marie, Technical Report March 1981 September 1983, CRDEC-TR-86041, "Engineering Development of the Simulator, Detector Tickets, Chemical Agent: Training M256 (TRAINS), June 1986, UNCLASSIFIED, Distribution B, U.S. Gov't Agencies Only.
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